ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[FRL–5267–2]

Guidelines Establishing Test Procedures for the Analysis of Pollutants; Chlorinated Pesticides and PCBs by Disk Extraction

AGENCY: Environmental Protection Agency (EPA).

ACTION: Final rule.

SUMMARY: This amendment to the Guidelines Establishing Test Procedures approves the use of an additional procedure for the determination of chlorinated pesticides and polychlorinated biphenyls (PCBs) in wastewater by adding appropriate citations to Tables IC and ID and by amending the incorporation by reference section of the regulation accordingly. The method differs from other approved methods in that it incorporates a disk of octadecyl-bonded silica enmeshed in a matrix of inert polytetrafluoroethylene (PTFE) fibrils for extraction of the analytes. The precision and recovery for the chlorinated pesticides and PCBs using this technique are not substantially different from those obtained using the liquid-liquid extraction technique already approved. Use of approved analytical techniques is required whenever the waste constituent specified is required to be measured for: an NPDES permit application; discharge monitoring reports; state certification; and other requests from the permitting authority for quantitative or qualitative effluent data. Use of approved test procedures is also required for the expression of pollutant amounts, characteristics, or properties in effluent limitations guidelines and standards of performance and pretreatment standards, unless otherwise specifically noted or defined.

DATES: This rule shall be effective on September 1, 1995. In accordance with 40 CFR 23.2 (45 FR 26048), these amendments to the regulation shall be considered issued for purposes of judicial review at 1 p.m. eastern time, August 16, 1995.

The incorporation by reference of certain publications listed in the regulation is approved by the Office of the Federal Register as of September 1, 1995.

Under section 509(b)(1) of the Clean Water Act, judicial review of these amendments can be obtained only by filing a petition for review in the United States Court of Appeals within 120 days after they are considered issued for purposes of judicial review. Under section 509(b)(2) of the Clean Water Act, these amendments may not be challenged later in civil or criminal proceedings brought by EPA to enforce these requirements.

FOR FURTHER INFORMATION CONTACT: Mr. James E. Longbottom, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268. Telephone number: (513) 569–7308.

SUPPLEMENTARY INFORMATION:

I. Authority

This regulation is promulgated under authority of sections 301, 304(h) and 501(a) of the Clean Water Act, 33 U.S.C. 1251 *et seq.* (the Federal Water Pollution Control Act Amendments of 1972 as amended) (the ''Act''). Section 301 of the Act prohibits the discharge of any pollutant into navigable waters unless the discharge complies with a National Pollutant Discharge Elimination System (NPDES) permit, issued under section 402. Section 304(h) of the Act requires the Administrator of the EPA to ''promulgate guidelines establishing test procedures for the analysis of pollutants that shall include the factors which must be provided in any certification pursuant to section 401 of this Act or permit application pursuant to section 402 of this Act''. Section 501(a) of the Act authorizes the Administrator to ''prescribe such regulations as are necessary to carry out his functions under this Act''.

II. Regulatory Background

The CWA establishes two principal bases for effluent limitations. First, existing discharges are required to meet technology-based effluent limitations. New source discharges must meet new source performance standards based on the best available demonstrated control technology. Second, where necessary, additional requirements are imposed to assure attainment and maintenance of water quality standards established by the States under Section 303 of the CWA. In establishing or reviewing NPDES permit limits, EPA must ensure that permitted discharges will not cause or contribute to a violation of water quality standards, including designated water uses.

For use in permit applications, discharge monitoring reports, and state certification and to ensure compliance with effluent limitations, standards of performance, and pretreatment standards, EPA has promulgated regulations providing nationally-approved testing procedures at 40 CFR Part 136. Test procedures have previously been approved for 262 different parameters. Those procedures apply to the analysis of inorganic (metal, non-metal, mineral) and organic chemical, radiological, bacteriological, nutrient, demand, residue, and physical parameters.

Additionally, some particular industries may discharge pollutants for which test procedures have not been proposed and approved under 40 CFR Part 136. Under 40 CFR Part 122.41 permit writers may impose monitoring requirements and establish test methods for pollutants for which no approved Part 136 method exists. 40 CFR 122.41(j)(4). EPA may also approve additional test procedures when establishing industry-wide technology-based effluent limitations guidelines and standards as described at 40 CFR 401.13.

The procedures for approval of alternate test procedures (ATPs) are described at 40 CFR 136.4 and 136.5. Under these procedures the Administrator may approve alternate test procedures for nationwide use which are developed and proposed by any person. 40 CFR 136.4 (a). Dischargers seeking to use such alternate test procedures on a limited basis (e.g., for their own discharge), must apply to the State or Regional EPA permitting office in which the discharge occurs approval under 136.4 (d). As specified below, today's rule approves an optional nationwide alternate procedure for determination of chlorinated pesticides and PCBs in wastewater test samples.

III. The Disk Extraction Test Procedure

The 3M Corporation, in accordance with the regulations published at 40 CFR section 136.5, applied for nationwide approval of their ''Organochlorine Pesticides and PCBs in Wastewater Using Empore Disk''. 3M subsequently presented data to meet the method comparability criteria set forth in the EPA ''Protocol for Approval of Alternate Test Procedures for Inorganic and Organic Analytes in National Pollutant Discharge Elimination System Monitoring'', July 12, 1993.

Extraction and concentration are preparation steps that are required prior to the determination of many organic analytes that are found in wastewater. The disk extraction procedure is proposed as an alternate to the presently approved liquid-liquid extraction procedure.

A. Scope of the Procedure

Method 3M 0222 is designed as an alternate test procedure for currently approved EPA Method 608. The Empore™ disk is used in place of liquid-liquid extraction. This method is being promulgated as an alternative procedure for the determination of nineteen specified organochlorine pesticides and seven PCBs listed below:

Aldrin
alpha-BHC
beta-BHC
Chlordane
delta-BHC
Dieldrin
Endosulfan I
Endosulfan II
Endosulfan sulfate
Endrin
Endrin aldehyde
gamma-BHC PCB-1254
Heptachlor
Heptachlor epoxide
Methoxychlor
Toxaphene
4,4′-DDD
4,4′-DDE
4,4′-DDT
PCB-1016
PCB-1221
PCB-1232
PCB-1242
PCB-1248
PCB-1254
PCB-1260

The parameters listed in the table can be determined by gas chromatography using Method 3M 0222. When the method is used to analyze unfamiliar samples for any or all of the compounds listed, compound identifications should be supported by at least one additional qualitative technique. The method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column.

B. Summary of the Methods

A measured volume of sample, approximately 1–L, is extracted using a 90 mm Empore™ disk. The disk is eluted with acetone followed by methylene chloride. The eluant is dried by pouring through anhydrous sodium sulfate and exchanged to hexane during concentration to a volume of 10-mL or less. The eluant is separated by gas chromatography and the analytes are then measured with an electron capture detector.

The method provides a Florisil column cleanup procedure and an elemental sulfur removal procedure using activated copper powder to aid in the elimination of interferences that may be encountered.

C. Technical Justification for Approved Procedure

The approval of this procedure is based on Agency review of the supporting information and data submitted by the applicant, 3M Corporation. EPA is approving the method based on the method description in EPA's Environmental Monitoring Management Council format, comparative analyses using the proposed and approved procedures, and EPA's technical and statistical reviews of each data package.

3M Corporation provided test data comparing the proposed procedure with appropriate approved procedure. The results from the proposed alternate method were compared to the approved EPA Method using liquid-liquid extraction/gas chromatography procedures. EPA statisticians and chemists conducted independent reviews of the data. The recovery and precision of all the submitted data for both the approved and proposed methods were also compared to the recovery and precision acceptance criteria derived for EPA Method 608 from Performance Evaluation Studies WP 18 and 23.

The Agency has judged the currently approved Method 608 method to be acceptable in the evaluation of the proposed procedure. EPA's Environmental Monitoring Systems Laboratory in Cincinnati, Ohio (EMSL-Cincinnati) thoroughly reviewed and evaluated the supporting data submitted by the 3M Corporation. The comparability reviews indicated that the analyses afforded comparable recovery and precision in the recommended concentration ranges for the listed organochlorine pesticides and PCBs. EPA proposed approval of the Empore™ disk procedure and sought public comment on the suitability of this method as an alternate procedure for use in the determination of the parameters listed in 59 FR 65878 (December 21, 1994). The administrative record is on file at EMSL-Cincinnati, 26 W. Martin Luther King Dr., Cincinnati, Ohio 45268. The record is available for public inspection. The approved procedure is also available from 3M Corporation, 3M Center Building 220–9E–10, St. Paul, MN 55144–1000.

Based on EMSL-Cincinnati's review, and pursuant to 40 CFR Section 136.5, EPA has approved the 3M Corporation's ''Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk'' method as an acceptable alternative procedure for nationwide use. Specifically, the method exhibits sufficient precision and recovery to establish (1) its acceptability under Part 136 and (2) its comparability to the approved procedure for analysis of the specified organochlorine pesticides and PCBs. As an approved alternate test procedure, this procedure is acceptable for use by any person required to test for these parameters.

IV. Public Comments and Response to Most Significant Comments

The Agency requested comments on the proposal to approve the 3M method for pesticides and PCB's. Comments were received from 5 individuals/organizations. All commenters favored approval of disk extraction as an acceptable alternate procedure (ATP). The most significant comments were as follows:

Comment: Other companies produce extraction disks on inert surfaces, so all references in the method to the disk in the 3M method should be generic in nature so that other commercial products can be used by the analyst. Commenter supports feasibility of generic approach by noting the method includes initial quality control demonstrations that can demonstrate applicability of the alternative vender's product, and that EPA used general product description language in the comparable method approved in 40 CFR 141 for drinking water analyses.

Response: EPA's limited resources are not sufficient to fully evaluate all new technologies that may be applicable to monitoring programs under the Clean Water Act. The nationwide alternate test procedure (ATP) program was established 40 CFR Part 136.4 to allow developers of new commercial instruments, product or supplies to demonstrate the efficacy of the measurement technology to measure pollutant concentration levels. The ATP program is expensive for the applicant as applicability to a broad variety of wastewaters must be demonstrated. The Agency does not require this applicant to demonstrate that the extraction technology can be made to work using competitor's products. The use of a competitive product in this method would require additional method development to optimize solvents, flow rates, and other features of the method. After these procedures have been standardized, a suitable demonstration of applicability is required. Because of the diverse nature of wastewaters under this regulation, a general statement of applicability could be made only if a number of different wastewaters are tested. Limited use approval could be obtained on a case-by-case basis by demonstrating applicability to an individual discharger's wastestream.

The quality control tests in the 3M method referenced by the commenter are performed using reagent water and will not demonstrate applicability to wastewater. The Agency actions in Part 141 were based on research on drinking water with commercial products from multiple suppliers. Since drinking waters do not contain the high organic loads and suspended solids that challenge the solid-phase extraction procedures, it is easier to establish general applicability to the matrix.

Comment: Commenter has tried these disks and has encountered some problems with plugging and finds no mention of what to do when this happens. Suggests method be limited to samples with less than 2–5% solids.

Response: In the comparison study performed by 3M, both the approved EPA Method 608 and the alternate 3M method produced lower results for wastewaters with very high suspended solids and the 3M method contains an appropriate caution in this regard. A sample with 2–5% solids is generally classified as a sludge and is beyond the scope of this rulemaking.

Comment: Commenter provided a series of questions for EPA to use in its evalation of the 3M method. The questions addressed technical specifications for the inert and active components of the disk, and possible limitations of the method caused by absorptive capacity, selective absorption or sample pH.

Response: The applicant voluntarily provided EPA with detailed responses to each of the questions, although much of this information would normally be treated by EPA as confidential business information. The applicant's response has been incorporated into the administrative record for this rulemaking. Alternate test procedures are evaluated primarily on the basis of method performance characteristics including accuracy, precision, and sensitivity data quality.

V. Regulatory Requirements

A. Executive Order 12866

Under Executive Order 12866, EPA must judge whether a regulation is ''major'' and, therefore, requires a regulatory impact analysis. EPA has determined that this regulation is not major as it will not result in an effect on the economy of $100 million or more, a significant increase in cost or prices, or any of the effects described in the Executive Order. This final rule would simply specify an alternative analytical procedure which may be used by laboratories in measuring concentrations of organochlorine pesticides and PCBs using EPA Method 608 and, therefore, would have no adverse economic impacts. This rule is not considered significant under the Executive Order.

B. Regulatory Flexibility Act

This amendment is consistent with the objectives of the Regulatory Flexibility Act (5 U.S.C. 602 et seq.) because it will not have a significant economic impact on a substantial number of small entities. The procedure included in this final rule would give all laboratories the flexibility to use this alternate procedure or not to use it.

C. Paperwork Reduction Act

This rule contains no requests for information activities and, therefore, no information collection request (ICR) was submitted to the Office of Management and Budget (OMB) for review in compliance with the Paperwork Reduction Act, (44 U.S.C. 3501 et seq.).

D. Unfunded Mandates

Under Section 202 of the Unfunded Mandates Reform Act of 1995, signed into law on March 22, 1995, EPA must prepare a written statement to accompany rules where the estimated costs to State, local, or tribal governments, or to the private sector will be § 100 million or more in any one year. Under Section 205, EPA must select the most cost-effective and least burdensome alternative that achieves the objective of such a rule and that is consistent with statutory requirements. Section 203 requires EPA to establish a plan for informing and advising any small governments that may be significantly and uniquely affected by the rule.

EPA estimates that the costs to State, local or tribal governments, or the private sector, from this rule will be far less than § 100 million. This rule should have minimal impact, if any, on the existing regulatory burden imposed on NPDES permittees required to monitor for regulated pollutants because the rule would merely make additional options available to the laboratory analyst conducting an existing approved test method. EPA has determined that an unfunded mandates statement therefore is unnecessary. Similarly, the method approved today does not establish any regulatory requirements that might significantly or uniquely affect small governments.

List of Subjects in 40 CFR Part 136

Environmental protection, Incorporation by reference, Water pollution control.

Dated: July 25, 1995.

Carol M. Browner,

Administrator.

In consideration of the preceding, EPA amends part 136 of title 40 Chapter I of the Code of Federal Regulations as follows:

PART 136—AMENDED

1. The authority citation for 40 CFR part 136 continues to read as follows:

Authority: Secs. 301, 304(h), 307, and 501(a) Public Law 95–217, Stat. 1566, *et seq.* (33 U.S.C. 1251 *et seq.*)(the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

2. Section 136.3 is amended as follows:

a. In Table 1C of paragraph (a) by revising entries 76. PCB–1016, 77. PCB–1221, 78. PCB–1232, 79. PCB–1242, 80. PCB–1248, 81. PCB–1254, 82. PCB–1260; and by adding footnote 8.

b. In Table ID of paragraph (a) by revising entries 1. Aldrin, 8. α-BHC, 9. β-BHC, 10. δ-BHC, 11. γ-BHC (Lindane), 15. Chlordane, 18. 4,4′-DDD, 19. 4,4′-DDE, 20. 4,4′-DDT, 28. Dieldrin, 32. Endosulfan I, 33. Endosulfan II, 34. Endosulfan sulfate, 35. Endrin, 36. Endrin aldehyde, 40. Heptachlor, 41. Heptachlor epoxide, 46. Methoxychlor, and 69. Toxaphene; and by adding footnote 8.

§ 136.3 Identification of test procedures.

(a) * * *

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter[1]	EPA method number[2 7]			Standard methods 18th ed.	ASTM	Other
	GC	GC/MS	HPLC			
* *	*	*	*	*	*	*
76. PCB–1016	608	625	.....	6410 B	.....	Note 3, p. 43; note 8.
77. PCB–1221	608	625	.....	6410 B	.....	Note 3, p. 43; note 8.
78. PCB–1232	608	625	.....	6410 B	.....	Note 3, p. 43; note 8.

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter [1]	EPA method number [2 7]			Standard methods 18th ed.	ASTM	Other
	GC	GC/MS	HPLC			
79. PCB–1242	608	625		6410 B		Note 3, p. 43; note 8.
80. PCB–1248	608	625				Note 3, p. 43; note 8.
81. PCB–1254	608	625		6410 B		Note 3, p. 43; note 8.
82. PCB–1260	608	625		6410 B, 6630 B		Note 3, p. 43; note 8.
* *	*		*	*	*	*

Table IC Notes

[1] All parameters are expressed in micrograms per liter (µg/L).

[2] The full text of Methods 601–613, 624, 625, 1624 and 1625, are given at appendix A, ''Test Procedures for Analysis of Organic Pollutants,'' of this part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at appendix B, ''Definition and Procedure for the Determination of the Method Detection Limit'' of this part 136.

[3] ''Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater,'' U.S. Environmental Protection Agency, September, 1978.

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[7] Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–613, 624, 625, 1624 and 1625 (See appendix A of the part 136) in accordance with procedures each in section 8.2 of each of these Methods. Additionally, each laboratory, on and on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for Methods 1624 land 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

[8] ''Organochlorine Pesticides and PCBs in Wastewater Using Empore TM Disk'', 3M Corporation Revised 10/28/94.

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES [1]

Parameter µg/L	Method	EPA [2 7]	Standard methods 18th ed.	ASTM	Other
1. Aldrin	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
* * *	*	*	*	*	*
8. α-BHC	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 8.
	GC/MS	[5] 625	6410 B		
9. β-BHC	GC	608	6630 C	D3086–90	Note 8.
	GC/MS	[5] 625	6410 B		
10. δ-BHC	GC	608	6630 C	D3086–90	Note 8.
	GC/MS	[5] 625	6410 B		
11. λ-BHC (Lindane)	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
* * *	*	*	*	*	*
15. Chlordane	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 8.
	GC/MS	625	6410 B		
* * *	*	*	*	*	*
18. 4,4′–DDD	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
19. 4,4′–DDE	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
20. 4,4′–DDT	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
* * *	*	*	*	*	*
28. Dieldrin	GC	608	6630 B & C		Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
* * *	*	*	*	*	*
32. Endosulfan I	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 8.
	GC/MS	[5] 625	6410 B		
33. Endosulfan II	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 8.
	GC/MS	[5] 625	6410 B		
34. Endosulfan Sulfate	GC	608	6630 C		Note 8.
	GC/MS	625	6410 B		
35. Endrin	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES [1]—Continued

Parameter µg/L	Method	EPA [2 7]	Standard methods 18th ed.	ASTM	Other
	GC/MS	[5] 625	6410 B		
36. Endrin aldehyde	GC	608			Note 8.
	GC/MS	625			
* * *	*		*	*	*
40. Heptachlor	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
41. Heptachlor epoxide	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 6, p. S73; note 8.
	GC/MS	625	6410 B		
* * *	*		*	*	*
46. Methoxychlor	GC		6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
* * *	*		*	*	*
69. Toxaphene	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
* * *	*		*	*	*

Table ID Notes:

[1] Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table 1C, where entries are listed by chemical name.

[2] The full text of Methods 608 and 625 are given at Appendix A. ''Test Procedures for Analysis of Organic Pollutants'', of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B. ''Definition and Procedure for the Determination of the Method Detection Limit'', of this Part 136.

[3] ''Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater'', U. S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.

[4] ''Methods for Analysis of Organic Substances in Water and Fluvial Sediments'', Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987).

[5] The method may be extended to include α-BHC, 1T1δ-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist in the sample, Method 608 is the preferred method.

[6] ''Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency''. Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 136) in accordance with procedures given in section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other Methods cited.

[8] ''Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk'', 3M Corporation, Revised 10/28/94.

3. In 136.3(b) the list entitled ''References, Sources, Costs, and Table Citations'' is amended by adding paragraph (33) to read as follows:

§ 136.3 Identification of test procedures.

* * * * *

(b) * * *

References, Sources, Costs, and Table citations:

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(33) ''Organochlorine Pesticides and PCBs in Wastewater Using Empore ™ Disk'' Test Method 3M 0222, Revised 10/28/94. 3M Corporation, 3M Center Building 220–9E–10, St. Paul, MN 55144–1000. Method available from 3M Corporation. Table IC, Note 8 and Table ID, Note 8.

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